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(54) Title of Invention Method for Preparing Crystalline Aluminosilicate

# Zeolite

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Specification

1 Title of the Invention

Method for Preparing Crystalline Aluminosilicate Zeolite

2. Claims

- (1) A method for preparing a ZSM-5 type crystalline aluminosilicate zeolite, comprising crystallizing a raw material mixture containing silica, alumina, an alkali metal, an organic cation, and water by heating, wherein upon crystallization, a crystalline aluminosilicate powder having the X-ray diffraction pattern shown in Table below, obtained by crystallizing a raw material mixture containing no organic cation, is allowed to be present in the raw material mixture.
- (2) The method as described in claim 1, wherein the crystalline aluminosilicate powder having the X-ray diffraction pattern shown in Table is present at 0.01 to 5% by weight of the raw material mixture.
- (3) The method as described in claim 1, wherein the crystalline aluminosilicate powder having the X-ray diffraction pattern shown in Table has a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 20 to 30.

3 Detailed Description of the Invention

[Industrial Availability]

The present invention relates to a method for preparing a ZSM-5 type crystalline aluminosilicate zeolite (which is hereinafter simply referred to as ZSM-5).

[Prior Art]

The ZSM-5 is synthesized by crystallizing a raw material mixture consisting of silica, alumina, alkali metals, tetrapropylammonia ions, and water according to Argauer, et al. (Japanese Patent Publication No. Showa 46-10064), and numerous methods for preparing the same have been proposed since.

A method for crystallization by allowing a seed crystal to be present in a raw

material mixture has been proposed. For example, the following methods can be mentioned.

- (1) The method as disclosed in Japanese Laid-Open Patent Publication No. Showa 56-37215 is a method of using a seed crystal so as to reduce the amount of an expensive organic mineralizer to be used.
- (2) The method as disclosed in Japanese Laid-Open Patent Publication No. Showa 57-7819 is a method for obtaining ZSM-5 by using a seed crystal instead of an organic mineralizer.

Any of these methods is aimed to reduce the amount of an organic mineralizer to be used. Further, the seed crystal in these methods is a crystal that is synthesized by allowing the organic mineralizer coexist, and even when this crystal is synthesized as a seed crystal, it is very difficult to obtain an average particle size of a final product of 1 micron or less, as described in detail later.

On the other hand, Japanese Laid-Open Patent Publication No. Showa 56-54222 and Japanese Laid-Open Patent Publication No. Showa 50-5335 each disclose a method for preparing ZSM-5 having a particle size of 1 micron or less, but these methods are methods which require thorough stirring upon crystallization or maintenance of a temperature of 90 to 110°C for 4 days before crystallization, and thus the processes are complicated.

[Problems to be Solved by the Invention]

It is an object of the present invention to provide a method for easily preparing ZSM-5 having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio and an average particle size of 1 micron or less on an industrial scale.

By decreasing the particle size, a catalyst having a very high catalytic activity and a long life time as a catalyst, when used as a catalyst for cracking, a catalytic conversion of a hydrocarbon, such as isomerization, and the like, can be obtained.

[Means for Solving the Problems]

The present inventors have found that ZSM-5 having a high silica ratio and a particle diameter of a final product of 1 micron or less is obtained by reacting a raw material mixture consisting of silica, alumina, alkali metals, organic cations, and water with a raw material mixture not containing an organic cation, and further, that it is easily obtained by allowing a crystalline aluminosilicate zeolite having an X-ray diffraction pattern as shown in Table 1 below to exist.

Table-1

Lattice plane spacing (Angstroms)		Relative strength
11.3	Strong	
10.0	Strong	
6.73	Weak	
6.41	Weak	
6.01	Weak	
5.73	Weak	
5.58	Weak	
5.01	Weak	
4.37	Weak	
4.28	Weak	
3.84	Very strong	
3.74	Strong	
3.66	Weak	
3.47	Weak	

3.37	Weak
3.32	Weak
3.06	Weak
2.99	Weak

The present invention has been made on the basis of this finding.

This seed crystal can be prepared, for example, in the manner to be described below. A raw material mixture, which includes white carbon as a silica source, sodium aluminate as an alumina source, and sodium hydroxide and water as an alkali conditioner, is prepared. Additionally, in a further method, a raw material mixture, in which an amorphous aluminosilicate homogeneous phase compound obtained by reacting an aqueous sodium silicate solution and an aqueous aluminum-containing solution is simultaneously or continuously dispersed in an aqueous sodium hydroxide solution, is prepared. It is a feature of the present invention that the raw material mixture has no organic cation. This raw material mixture has a composition as shown in terms of each oxide molar ratio:

$$SiO_2/Al_2O_3 = 20 \text{ to } 45$$

$$Na_2O/SiO_2 = 0.05$$
 to 0.3

$$H_2O/SiO_2 = 12$$
 to 70.

After stirring the raw material mixture at room temperature until it turns sufficiently homogeneous, it is kept at a temperature of 120°C to 200°C for 5 hours to 5 days under stirring in an autoclave to carry out crystallization. Next, it is subjected to solid-liquid separation, sufficiently washed, and dried at 110°C overnight.

The crystalline zeolite thus obtained is a crystalline zeolite having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 20 to 30 and the X-ray diffraction pattern shown in Table 1, which would become a seed crystal usable in the present invention.

It is one of the features of the X-ray diffraction pattern of the seed crystal that although synthesis is conducted without the use of an organic mineralizing agent, only two peaks are observed between the lattice plane spacing d=3.70 Å and d=3.86 Å. For ZSM-5 which is synthesized with the addition of organic mineralizing agent, 2 to 3 peaks are observed between the lattice plane spacing d=3.70 Å and d=3.86 Å, as disclosed in Japanese Patent Publication No. 46-10064. However, for the X-ray diffraction pattern of the ZSM-5-like zeolite which is synthesized with no addition of an organic mineralizing agent and with the use of silica sol as a silica source, as disclosed in Japanese Patent Publication No. 56-49851, 4 peaks are observed between the lattice plane spacing d=3.70 Å and d=3.86 Å. That is, the seed crystal used in the present invention is a crystalline aluminosilicate zeolite which is completely different in nature from the ZSM-5-like zeolite as disclosed in Japanese Patent Publication No. 56-49851.

The ZSM-5 type crystalline aluminosilicate zeolite which is a seed crystal used in the present invention, which is synthesized without using the organic mineralizer as described above, and which has an X-ray diffraction pattern shown in Table 1, is characterized by having a crystal particle diameter as small as hundreds of Angstroms and small agglomerates, as compared to other ZSM-5 or ZSM-5-like zeolites. The ZSM-5 type crystalline aluminosilicate zeolite as a seed crystal may be obtained as an agglomerate of relatively large crystal particles in some cases, but this agglomerate has a very weak bond between the crystal particles. Thus, it can be easily made into small particles by dispersing it in water or by milling it in a mill. This is also a significant characteristic of the seed crystal of the present invention.

On the other hand, the ZSM-5 type and ZSM-5-like zeolites as disclosed hitherto have large crystal particles, and if it is provided as an agglomerate, the bond between the crystal particles of the agglomerate is strong, and accordingly, it is very

difficult to make it small particles, even by dispersing it in water or milling it in a mill. Even by using it as a seed crystal, it is apparent that the ZSM-5 having a size of 1 micron or less cannot be prepared.

The crystalline zeolite having an X-ray diffraction pattern as shown in Table 1, obtained by crystallizing a raw material mixture not containing an organic cation, has a  $SiO_2/Al_2O_3$  molar ratio of 15 to 35.

If the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio is more than 35, the particle diameter becomes very large, and further, the shape of the particle is completely different from those having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 20 to 30. That is, the seed crystal used in the present invention, having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 20 to 30, has a small crystal particle, and if provided as an agglomerate, can be transformed from a relatively large agglomerate to a small particle, by a simple method, for example, by being dispersed in water or by being milled in a mill.

The figure shows an electronic microscope photo of the seed crystal used in the present invention. As seen from the picture, the particle of the seed crystal is an agglomerate formed by the collection of small crystals without a clear outline of the particle, which can be easily milled as described above.

If ZSM-5 is synthesized using the seed crystal according to the present invention having the characteristics as above, ZSM-5 having a particle diameter of 0.1 to 1.0 micron is obtained. Additionally, surprisingly, the nucleation of ZSM-5 occurs vigorously on the particle surface of the seed crystal, and the nucleus grows only to a size of at most 0.5 micron. In addition, it could be seen that the seed crystal did not grow at all. Moreover, it was also surprising that the seed crystal and the particle of ZSM-5 are separated by a later washing step, which thus leads to homogeneous ZSM-5 having a particle diameter of 1 micron or less.

On the other hand, if synthesis is carried out using common ZSM-5 or ZSM-5-like zeolites as disclosed in Japanese Patent Publication No. Showa 46-10064 or Japanese Patent Publication No. Showa 56-49851 as a seed crystal, a large seed crystal that cannot be milled remains in the product, and in addition, the seed crystal causes the crystal growth, and as a result, a product admixed with a very large particle is obtained and the nucleation on the surface of the seed crystal particle is significantly small.

Therefore, the seed crystal according to the present invention has completely different properties from those of general ZSM-5 or ZSM-5-like zeolites. That is, the seed crystal which can be used in the present invention has the X-ray diffraction pattern shown in Table 1, which is obtained by the crystallization of the raw material mixture not containing organic cation. There is only a crystalline aluminosilicate powder having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 15 to 35. The seed crystal is allowed to be present at 0.01 to 5% by weight of the raw material mixture.

The silica source used in the present invention is not particularly limited as long as it is one usually used for the preparation of a crystalline zeolite, and examples thereof include white carbon, colloidal silica, fused silica, and the like.

The alumina source used in the present invention is not particularly limited as long as it is one usually used for the preparation of a crystalline zeolite, and examples thereof include sodium aluminate, aluminum sulfate, potassium aluminate, aluminum nitrate, aluminum chloride, colloidal alumina, alumina, and the like.

Further, as the silica source and the alumina source, a homogeneous amorphous aluminosilicate compound, zeolite, or the like can also be used.

As the alkali metal source, sodium hydroxide, potassium hydroxide, or the like, as well as sodium silicate which also serves as a silica source, sodium aluminate which also serves as an alumina source, or the like is used.

The organic cation is not particularly limited as long as it is one of those usually used for the ZSM-5 type or ZSM-5-like zeolite, and examples thereof include tetraalkylammonium ions, alkylamine, ammonia ions, and the like.

As the conditions such as the ratio of raw materials, the temperature, the pressure, the time, and the like, ones that are usually used can be used.

It is preferable that the ratio of silica to alumina in the raw material mixture of the present invention is an  $SiO_2/Al_2O_3$  molar ratio of 10 to  $\infty$ , and the ratio of the alkali metals (M) to silica is an  $M_2O/SiO_2$  molar ratio in the range of 0.01 to 30, and the ratio of the organic cation (R) to silica is an  $R_2O/SiO_2$  molar ratio in the range of 0.0001 to 10. In addition, the seed crystal is present at 0.01 to 5% by weight of the raw material mixture.

The synthesis reaction of the present invention is carried out by using the above-described raw material mixture in an autoclave at 80°C to 300°C and at an autogenous pressure until a crystalline zeolite is produced. The time taken for the crystallization varies depending on a heating temperature, but it is in a range from 1 hour to 100 days.

After completion of the crystallization, the product is sufficiently washed with water and dried in well-known methods to obtain crystal powders. The particle of the crystal thus obtained is a ZSM-5 type crystalline zeolite having an average particle size of 1 micron or less and a substantially uniform particle size distribution.

[Effect of the Invention]

As seen from the above description, according to the present invention, a high-purity ZSM-5 having an average particle size of 1 micron or less and a high SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio can be easily obtained.

The ZSM-5 obtained by the present invention may be subjected to ion exchange

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with appropriate cations, and to sintering, if necessary, and then it can be used as an adsorbent, a molecular sieve, and a catalyst.

[Examples]

<Pre><Preparation of Seed Crystal>

Seed crystal A

850 g of pure water, 6.0 g of solid sodium hydroxide, and 51 g of an aqueous sodium aluminate solution (Na<sub>2</sub>O: 18.6 wt%, Al<sub>2</sub>O<sub>3</sub>: 20.0 wt%, H<sub>2</sub>O: 61.4 wt%) were mixed to give a uniform aqueous solution, and then 170 g of white carbon (manufactured by Nippon Silica Co., Tradename: Nipsil VN-3, SiO<sub>2</sub>: 87.7 wt%, Al<sub>2</sub>O<sub>3</sub>: 0.5 wt%) was added thereto under stirring. This raw material mixture was crystallized in an autoclave at 160°C for 72 hours, and the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at 110°C. Then, the product was pulverized in a mortar. The resulting product had the X-ray diffraction pattern as shown in Table 2. The results of the chemical analysis were 1.1Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·21.1SiO<sub>2</sub>.

Table 2

| Lattice plane spacing (Angstroms) |    | Relative strength |
|-----------------------------------|----|-------------------|
| 11.3                              | 50 |                   |
| 10.0                              | 41 |                   |
| 6.73                              | 9  |                   |
| 6.41                              | 8  |                   |
| 6.01                              | 12 |                   |
| 5.73                              | 9  |                   |
| 5.58                              | 10 |                   |

| 5.01 | 9   |
|------|-----|
| 4.37 | 8   |
| 4.28 | 8   |
| 3.84 | 100 |
| 3.74 | 57  |
| 3.66 | 25  |
| 3.47 | 10  |
| 3.37 | 11  |
| 3.32 | 12  |
| 3.06 | 12  |
| 2.99 | 19  |

# Seed crystal B

An aqueous silicate soda solution (SiO<sub>2</sub>: 25 wt%, Na<sub>2</sub>O: 8.2 wt%) at a feeding rate of 1.5 l/hr and an aqueous aluminum sulfate solution (Al<sub>2</sub>O<sub>3</sub>: 4.3 wt%, SO<sub>4</sub>: 33.3 wt%) added with sulfuric acid at a feeding rate of 0.5 l/hr were fed into an overflow type reaction tank under stirring (real capacity: 5.5 l) simultaneously and continuously to carry out a reaction.

The apparent retention time of the reaction slurry was 30 minutes, the reaction temperature was from 30 to 32°C, and the pH of the overflowing slurry was from 6.3 to 6.6. The discharged slurry was subjected to solid-liquid separation, and then sufficiently washed with water to obtain a homogeneous amorphous aluminosilicate compound having the composition of 1.1Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·26.1SiO<sub>2</sub>·140H<sub>2</sub>O.

266 g of the homogeneous amorphous aluminosilicate compound thus prepared was added to an aqueous solution obtained by mixing 426 g of pure water and 7.1 g of

solid sodium hydroxide under stirring. This raw material mixture was crystallized in an autoclave at  $155^{\circ}$ C for 72 hours, and the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at  $110^{\circ}$ C. Then, the product was pulverized in a mortar. The resulting product had substantially the same X-ray diffraction pattern as in Table 2. The results of the chemical analysis were  $1.0Na_2O\cdot Al_2O_3\cdot 22.4SiO_2$ .

# Seed crystal C

415 g of pure water, 8.0 g of solid sodium hydroxide, and 6.6 g of sodium aluminate (Al<sub>2</sub>O<sub>3</sub>: 32.98 wt%, Na<sub>2</sub>O: 35.13 wt%) were mixed to give a uniform aqueous solution, and then 270 g of a colloidal silica sol (manufactured by Shokubai Kasei Kogyo K.K, Tradename: Cataloid S-30L, SiO<sub>2</sub>: 30 wt%) was added thereto under stirring. This raw material mixture was crystallized in an autoclave at 180°C for 21 hours, and the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at 110°C. Then, the product was pulverized in a mortar.

The results of the X-ray diffraction of the product were substantially the same as in Table 2, except that four peaks appeared between d=3.70 Angstroms and d=3.86 Angstroms. The results of the chemical analysis were 1.0Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·42SiO<sub>2</sub>.

# Seed crystal D

487 g of pure water, 10.6 g of solid sodium hydroxide, 23.7 g of tetrapropylammonium bromide, and 2.8 g of an aqueous sodium aluminate solution (Na<sub>2</sub>O: 18.6 wt%, Al<sub>2</sub>O<sub>3</sub>: 20.0 wt%, H<sub>2</sub>O: 61.4 wt%) were mixed to give a uniform aqueous solution, and then 76.1 g of white carbon (manufactured by Nippon Silica Co.,

Tradename: Nipsil VN-3, SiO<sub>2</sub>: 87.7 wt%, Al<sub>2</sub>O<sub>3</sub>: 0.5 wt%) was added thereto under stirring. This raw material mixture was crystallized in an autoclave at 160°C for 48 hours, and the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at 110°C. Then, the product was pulverized in a mortar. The resulting product had substantially the same X-ray diffraction pattern as in Table 2. The results of the chemical analysis showed that the product was 1.5Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·103SiO<sub>2</sub>.

### Seed crystal E

580 g of pure water, 5.9 g of solid sodium hydroxide, 28.7 g of tetrapropylammonium bromide, and 20.5 g of an aqueous sodium aluminate solution (Na<sub>2</sub>O: 18.6 wt%, Al<sub>2</sub>O<sub>3</sub>: 20.0 wt%, H<sub>2</sub>O: 61.4 wt%) were mixed to give a uniform aqueous solution, and then 92.2 g of white carbon (manufactured by Nippon Silica Co., Tradename: Nipsil VN-3, SiO<sub>2</sub>: 87.7 wt%, Al<sub>2</sub>O<sub>3</sub>: 0.5 wt%) was added thereto under stirring. This raw material mixture was crystallized in an autoclave at 160°C for 48 hours, and the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at 110°C. Then, the product was pulverized in a mortar. The resulting product had substantially the same X-ray diffraction pattern as in Table 2. The results of the chemical analysis showed that the product was 1.1Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·27SiO<sub>2</sub>.

### Example 1

487 g of pure water, 10.6 g of solid sodium hydroxide, 23.7 g of tetrapropylammonium bromide, and 2.8 g of an aqueous sodium aluminate solution (Na<sub>2</sub>O: 18.6 wt%, Al<sub>2</sub>O<sub>3</sub>: 20.0 wt%, H<sub>2</sub>O: 61.4 wt%) were mixed to give a uniform

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aqueous solution, and then 76.1 g of white carbon (manufactured by Nippon Silica Co., Tradename: Nipsil VN-3, SiO<sub>2</sub>: 87.7 wt%, Al<sub>2</sub>O<sub>3</sub>: 0.5 wt%) was added thereto under stirring. To this raw material mixture was added 3 g of crystalline aluminosilicate powder (seed crystal A), and then the mixture was put into a 1-liter autoclave, followed by stirring a peripheral velocity of 0.8 m/sec and heating at 180°C for 14 hours, thereby carrying out crystallization. Then, the reaction product was subjected to solid-liquid separation, sufficiently washed with water, and then dried at 110°C. The results of the X-ray diffraction of the product are shown in Table 3. The results of the chemical analysis showed that the product was 1.1Na<sub>2</sub>O·Al<sub>2</sub>O<sub>3</sub>·89SiO<sub>2</sub>. This product had an average particle size of 0.8 microns.

Table 3

| Lattice plane spacing (Angstroms) |    | Relative strength |
|-----------------------------------|----|-------------------|
| 11.2                              | 42 |                   |
| 10.0                              | 29 |                   |
| 7.50                              | 8  |                   |
| 6.70                              | 5  |                   |
| 6.37                              | 9  |                   |
| 6.03                              | 10 |                   |
| 5.73                              | 7  |                   |
| 5.57                              | 8  |                   |
| 5.00                              | 6  |                   |
| 4.62                              | 7  |                   |
| 4.37                              | 8  |                   |
| 4.26                              | 11 |                   |

| 4.00 | 8   |
|------|-----|
| 3.83 | 100 |
| 3.72 | 44  |
| 3.65 | 27  |
| 3.44 | 10  |
| 3.32 | 10  |
| 3.05 | 10  |
| 2.98 | 12  |

# Example 2

The procedure was carried out in accordance with Example 1, except that the amount of the crystalline aluminosilicate powder (seed crystal A) to be added was 18.0 g. The results of the X-ray diffraction of the product were substantially the same as in Table 3. Further, the results of the chemical analysis showed that the product was  $0.91Na_2O\cdot Al_2O_3\cdot 57SiO_2$ . This product had an average particle size of 0.7 microns.

# Example 3

The procedure was carried out in accordance with Example 1, except that 3.0 g of the seed crystal B was added instead of the seed crystal A as a crystalline aluminosilicate powder. The results of the X-ray diffraction of the product were substantially the same as shown in Table 3. Further, the results of the chemical analysis were  $1.1Na_2O\cdot Al_2O_3\cdot 91SiO_2$ . This product had an average particle size of 0.8 microns.

# Comparative Example 1

The procedure was carried out in accordance with Example 1, except that the crystalline aluminosilicate powder was not added to the raw material mixture. The results of the X-ray diffraction of the product were substantially the same as in Table 3. This product had an average particle size of 2.3 microns.

# Comparative Example 2

The procedure was carried out in accordance with Example 1, except that 18.0 g of the seed crystal C was added instead of the seed crystal A as a crystalline aluminosilicate powder. This product had an average particle size of 3.0 microns.

# Comparative Example 3

The procedure was carried out in accordance with Example 1, except that 18.0 g of the seed crystal D was added instead of the seed crystal A as a crystalline aluminosilicate powder. This product had an average particle size of 1.8 microns.

# Comparative Example 4

The procedure was carried out in accordance with Example 1, except that 18.0 g of the seed crystal E was added instead of the seed crystal A as a crystalline aluminosilicate powder. This product had an average particle size of 2.2 microns.

# 4 Brief Description of Drawings

The figure shows an electronic microscope photo of one example of the seed crystals used in the present invention.

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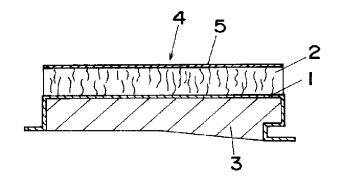
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### (54) 【発明の名称 】 浴室壁材

### (57)【要約】

【目的】 熱による木質材の寸法変化を抑制する。

【構成】 ひびが入れられた後に樹脂含浸処理された木 質材2を浴室用基材1に積層する。木質材1と浴室用基 材1との熱膨張率の差が縮まる。浴室内の熱気が木質材 2に加わっても、浴室壁材4のバイメタル現象が抑制さ れる。反り、変形等を防止できる。



浴室用基材 浴室壁材

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### 【発明の詳細な説明】

### [0001]

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### [0002]

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### [0003]

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【0004】本発明は、上記従来の課題に鑑みてなされたもので、その目的とするところは、熱による木質材の寸法変化を抑制することにより、反り、変形等を防止できるようにした浴室壁材を提供するにある。

### [0005]

【課題を解決するための手段】上記課題を解決するため、本発明は、ひびが入れられた後に樹脂含浸処理された木質材2を浴室用基材1に積層したことを特徴とする。

### [0006]

【作用】本発明によれば、木質材2に樹脂含浸処理に先立ってひび入れ処理を施すものであって、こうして得られた木質材2を浴室用基材1に積層するようにしたので、浴室内の熱気が木質材2に加わると、木質材2に含まれていた水分が飛んで収縮し、浴室用基材1との熱膨張率の差、及び水分の吸・脱着による膨潤、収縮の差が縮められる。従って、熱又は水による浴室壁材4のバイメタル現象が抑制され、幅方向の反り量を減少させることができる。

# [0007]

【実施例】以下、本発明の一実施例を図面に基づいて説明する。本実施例に用いられる浴室壁材4は、図1に示すように、浴室用基材1(バスリブ)と木質材2(単板)とが積層されて成る。図中、3はウレタン発泡体である。上記木質材2は、樹脂含浸処理に先立ってテンダライズイング(ひびを入れる)されており、木質材2の

寸法変化を抑制でき、浴室壁材4のバイメタル現象が抑 えられるようになっている。

【0008】浴室壁材4の作製方法の一例として、まず、木質の単材(1.0tラワン)をテンダライザーにて切り刻んでひび入れ加工する。この単板に接着剤を用いて木質のスライス単板5(0.2t)を貼り加工する。なお、ひびの深さ、ピッチは条件を設定しても材料により異なることから、単板の材質に応じたひびの深さ、ピッチの条件に設定しておく。こうして得られた木質材2に、従来工法により、樹脂含浸処理(PM処理)を行った後に、浴室用基材1(バスリブ)に貼り加工、溝加工、塗装加工を施して製品化する。かかる製品4は熱又は水による幅方向の反り量が1.0mm~2.0mmから、02mm以下となることが認められた。また、木質材2をテンダライズイングしたことにより樹脂含浸処理の含浸率が10%~15%向上することが確認された。

【0009】上記のように、木質材2に樹脂含浸処理に 先立ってひび入れ処理を施し、こうして得られた木質材 2を浴室用基材1に積層するようにしたので、浴室内の 熱気が浴室壁材4に加わると、浴室用基材1が塩化ビニ ルの場合は、この塩化ビニルは熱を加えると伸びるが、 テンダライズイングした木質材2は熱を加えると含まれ ていた水分が飛んで収縮し、浴室用基材1との熱膨張率 の差、及び水分の吸・脱着による膨潤、収縮の差が縮め られる。その結果、熱又は水による浴室壁材4のバイメ タル現象が抑制され、その幅方向の反り量が減少するこ とにより、反り、変形等の発生を防止できるのである。 しかも、木質材2にひび入れ処理を施したことにより、 樹脂含浸処理の含浸率が向上し、木質材2の含浸速度を 速めて生産性が向上するという利点もある。

### [0010]

【発明の効果】本発明は上述のように、ひびが入れられた後に樹脂含浸処理された木質材を浴室用基材に積層したから、浴室壁材のバイメタル現象を抑制して、反り、変形等を防止できると共に、木質材にひび入れ処理を施したことによって、樹脂含浸処理の含浸率が向上する。その結果、高品質で且つ生産性に優れた浴室壁材を得ることができるという効果がある。

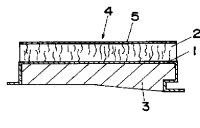
### 【図面の簡単な説明】

【図1】本発明の一実施例の断面図である。

### 【符号の説明】

- 1 浴室用基材
- 2 木質材
- 4 浴室壁材

【図1】



1 浴室用基材 2 木質材 4 浴室壁材